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Microspheres for Laser Velocimetry in High Temperature Wind Tunnel

by

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The introduction of non-intrusive measurement techniques in wind tunnel experimentation has been a turning point in error free data acquisition. Laser velocimetry has been progressively implemented and utilized in various wind tunnels; e.g. subsonic, transonic, and supersonic. The success of the laser velocimeter technique is based on an accurate measurement of scattered light by seeding particles introduced into the flow stream in the wind tunnel. Therefore, application of appropriate seeding particles will affect, to a large extent the acquired data. The seeding material used depends on the type of experiment being run. Among the seeding material for subsonic tunnel are kerosene, Kaolin, and polystyrene. Polystyrene is known to be the best because of being solid particles, having high index of refraction, capable of being made both spherical and monodisperse. However for high temperature wind tunnel testing seeding material must have an additional characteristic that is high melting point. Typically metal oxide

Powders such as Al_2O_3 with melting point $3660^{\circ}F$ is being used. The metal oxides are however polydispersed, have a high density, and a tendency to form large agglomerate that does not closely follow the flow velocity. The addition of flame phase silica to metal oxide helps to break up the agglomerates, however, still results in a narrow band of polydispersed seeding. The less desirable utility of metal oxide in high temperature wind tunnels necessitates the search for a better alternative particle seeding which this paper addresses.

The Laser Velocimetry (L. V.) characteristic of polystyrene makes it a prime candidate as a base material in achieving the high temperature particle seeding inexpensively. While polystyrene monodisperse seeding particle reported (1-2) has been successful in subsonic wind tunnel, but lacks the high melting point and thus is not practically usable in high temperature wind tunnel.

It is well known that rise in melting point of polystyrene can be achieved by cross-linking technique(3). Since polystyrene already possesses all the desired characteristics for LV, to circumvent the low melting point, cross-linking technique was investigated.

CROSS-LINKED POLYSTYRENE PREPARATION

The ingredients in mixture are water as medium, magnesium sulfate electrolyte, styrene monomer, divinylbenzene (DVB) as cross-linking agent, and potassium Persulfate as initiator. The lack of established formulations for mixture opens the way for trial and error process. Therefore a polymerization process was chosen as starting point as follows:

1. A water bath filled with tap water was heated to reach 65°C, a pyrex reaction kettle was filled with 2370 (ml) high purity distilled water, 56 (ml) magnesium sulfate, 265 (ml) styrene and a volume of divinylbenzene as part of styrene volume. An agitator is placed in the reactor before it was covered.
2. The reactor is placed in the water bath for 40 minutes. During this period nitrogen gas is flowing through the mixture to purge oxygen with the agitator turning at a rate of 150 rpm.
3. Potassium Persulfate solution was added to the reactor at 65°C.
4. For cross-linking to take place, the mixture was run for 18 hours. At the end of this period the reactor was removed from the water bath and filtered through 100 mesh cheese cloth into a clean storage container.

The simplicity and very low cost of producing the seeding particles make this process, practically a "do-it-yourself" manufacturing, which is very attractive when compared to the commercial market of several hundreds of dollars per pound. However, this process is still in developing stages, and has a long way to be transferred from a trial and error process to a straight forward, well-established process. A well-established process requires known formulation of material solution for desired particle size in conjunction with detail specification of ever changing environmental parameters. The trial and error nature of this procedure require a large number of experimentation therefore twenty-four hours run were adopted. In general rate of success was 12.5% that is relatively good as first set of experimentation considering high number of affecting factors. They include five material solutions; water, magnesium sulfate, styrene, divinylbenzene, potassium persulfate, and at least five environmental factors that controls the condition in which beads are being produced: temperature of mixture, rotation rate of agitator, rate of flow of nitrogen into the mixture, kind and placement of the stirrer, and required time for desired bead size.

RESULTS

The trial and error nature of this process were the cause of many surprises and produced from very thin transparent latex sheets' pieces too soft and delicate to snow like white flacks, and to golf ball size styrene spheres.

Early in the experimentation it was decided to keep all the parameters the same and vary the amount of divinylbenzene. The volume amount of divinylbenzene (DVB) has been varied from 5% to 0.1% of styrene volume. Experimentation using DVB 2% of styrene volume produced some beads, figure 1, while a close up view is shown in figure 2. In order to determine the melting point a sample was tested in Differential Scanning Calorimeter (DSC), figure 3, which illustrates there is not a melting point in the temperature range tested and in fact cross-linking has been accomplished even though at temperature of 103.61°C it has a phase two change. At this temperature the white sample changes to brown, at which it stays throughout until it decays. The temperature range of 0°C to 300 °C is the expected range of temperature in the wind tunnel. For comparison proposes, a sample of polystyrene was prepared and tested in DSC, figure 4, which indicates melting temperature of 193°C. Even though this run showed that the cross-linking is possible, the beads were too attached and crumbled together. They form a worm like shape even though among them existed individual spherical beads. Hence

the batch was generally not usable. To clarify this point a sample of non cross-linked polystyrene is shown in figure 5, which illustrates the polystyrene beads are monodisperse spherical shape of $0.95\ \mu$ diameter, on average.

Even though all the parameters except for DVB were kept constant, the results of the runs were not similar and at times there were not any beads produced at all or products existed in worm like form. This situation made most of the batches not usable. Another aspect of this process is the time it takes to finish a run, namely 18 hours on average, which limits the number of the runs that could be made. The reason for all this inconsistencies is related to fluctuations of environmental parameters, even though care was taken to keep them constant as well as it could be done. Affect of the factors such as vibration of bath tub, outside temperature etc. contributed to these fluctuation.

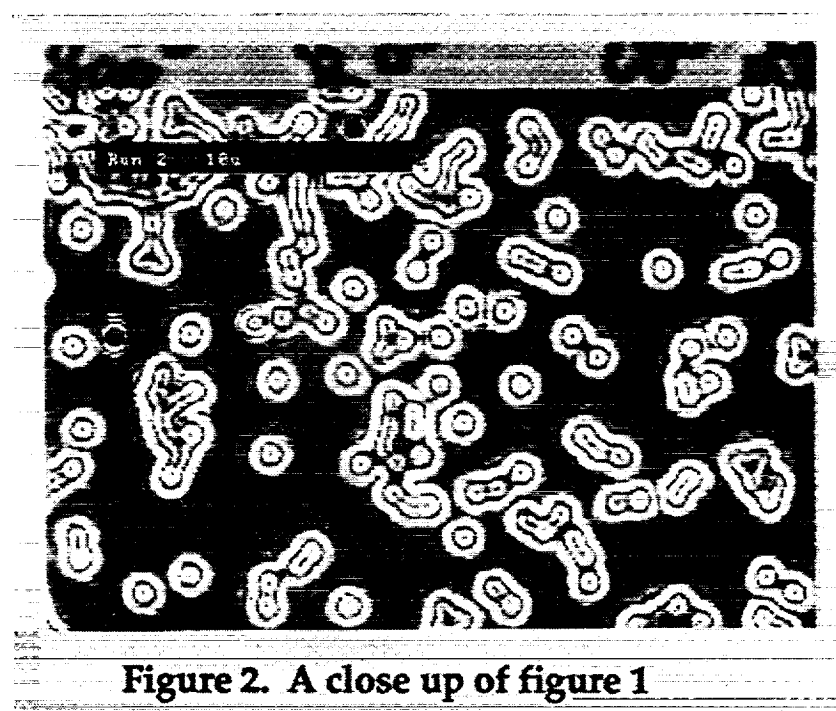
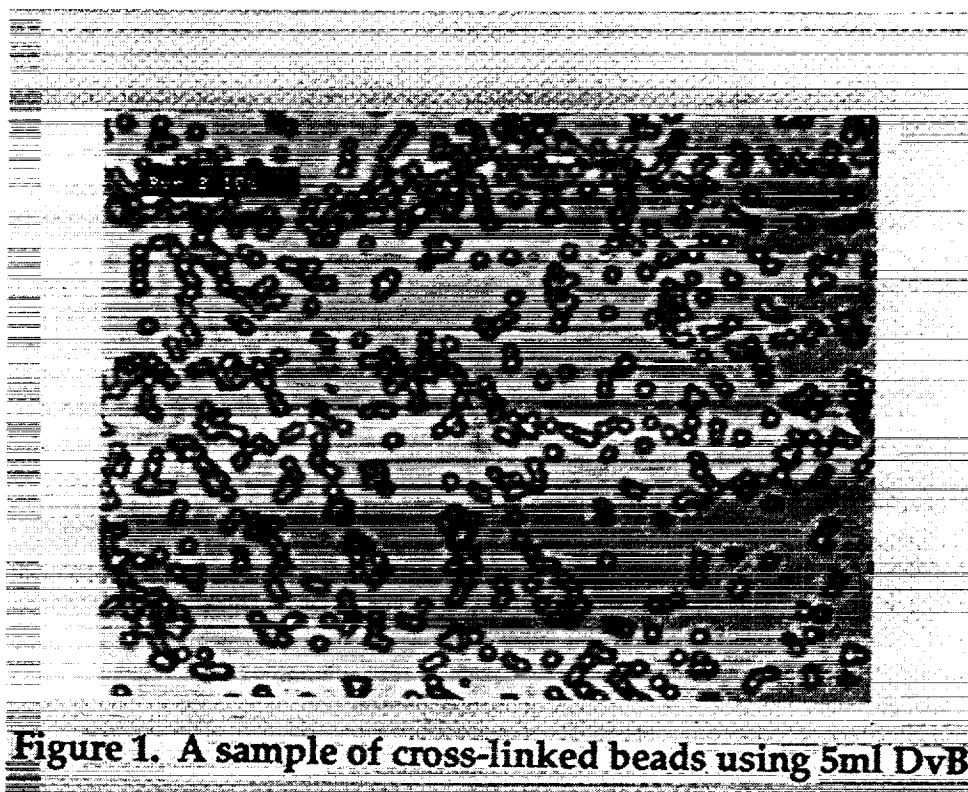
A major surprise was a run with DVB volume only 0.2% of styrene volume. The run was monitored every 2 hours for the first and last 8 hours. The sample taken after 4 hours clearly showed that beads were formed, figure 6. This figure indicates that beads are spherical. A computer based particle sizing technique was used to determine the size of the seeding spheres. Figure 7 demonstrates the monodispersity of the beads with average diameter of $0.95\ \mu$. This run was sampled after 22 hours and contrary to forming larger and better product, there were only a few good beads shown distinctively, the rest being much smaller beads crumbled together or overlaid each other which made the result of this run an unusable batch, figure 8. For comparing purposes this run was repeated with exact amounts and monitored at the same intervals. Surprisingly, the 4 hours sample did not show any formulation of beads, figure 9. The 6 hours sample showed worm like beads formation, figure 10. The 19 hours sample illustrated the formation of beads, figure 11, however not as good as expected. The monodispersity of this sample is illustrated in figure 12. The beads are of size $0.7\ \mu$ and monodisperse. In general last two aforementioned runs indicate how sensitive the process is to unforeseen change.

CONCLUSION

It was experimentally demonstrated that cross-linking of polystyrene does produce seeding particle for high temperature wind tunnel. Indeed among several runs that were made a few batch resulted in spherical beads with no melting point in the desired range of 0°C to 300°C . Considering the cost of the commercially available beads (a few dollar in oppose to hundreds of dollars per ten grams), good L. V. characteristics, and lightness in compare with metal oxides, the cross-linked polystyrenes particles makes a superior seeding candidate for high temperature and low temperature wind tunnels.

REFERENCES

1. Nichols, Jr., C.E., "Preparation of Polystyrene Microspheres for Laser Velocimetry in Wind Tunnel", NASA Technical Memorandum, 89163, Jun(1987).
2. Ghorieshi, J. A., "Wind Tunnel Seeding Particles for Laser Velocimetry", NASA publication, Hampton, VA(1992).
3. Personal conversation with Warren C. Kelliher, Facility Engineering Division, NASA, Hampton, VA(1993)



Sample: X-LINKED #1
Size: 4.0000 mg
Method: RT-300 20°C/MIN

DSC

File: 1341.01
Operator: KAREN WHITLEY
Run Date: 06/21/93 09:24

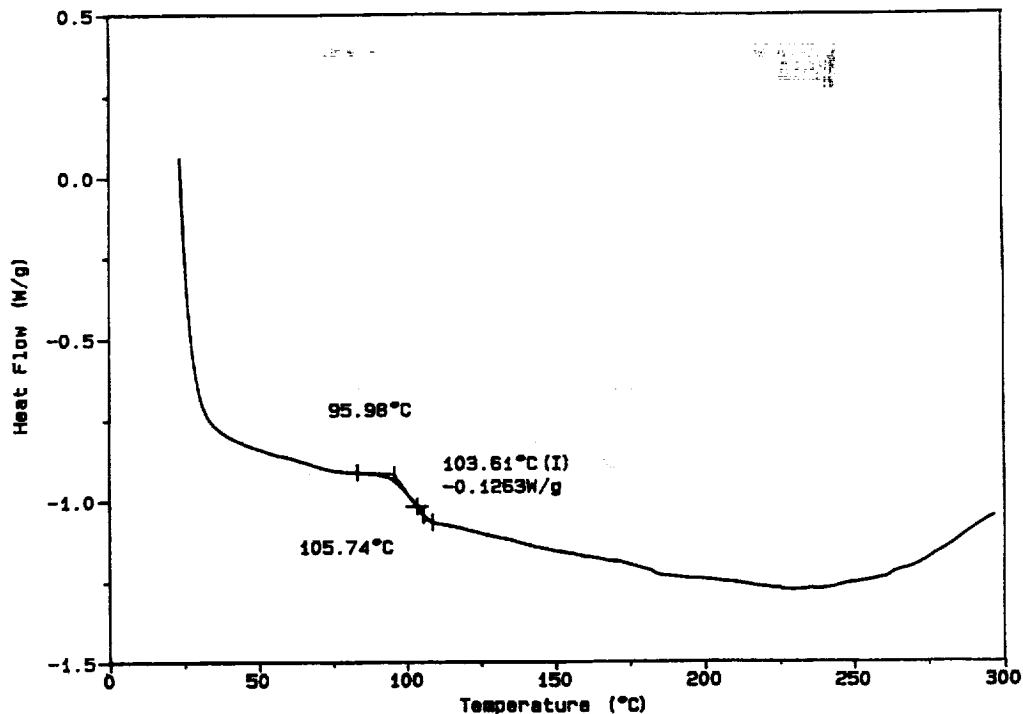


Figure 3. Cross-linked polystyrene temperature variation curve indicating no melting point

Sample: POLYSTYRENE
Size: 4.8000 mg
Method: RT-300 20°C/MIN

DSC

File: 1340.01
Operator: KAREN WHITLEY
Run Date: 06/18/93 14:51

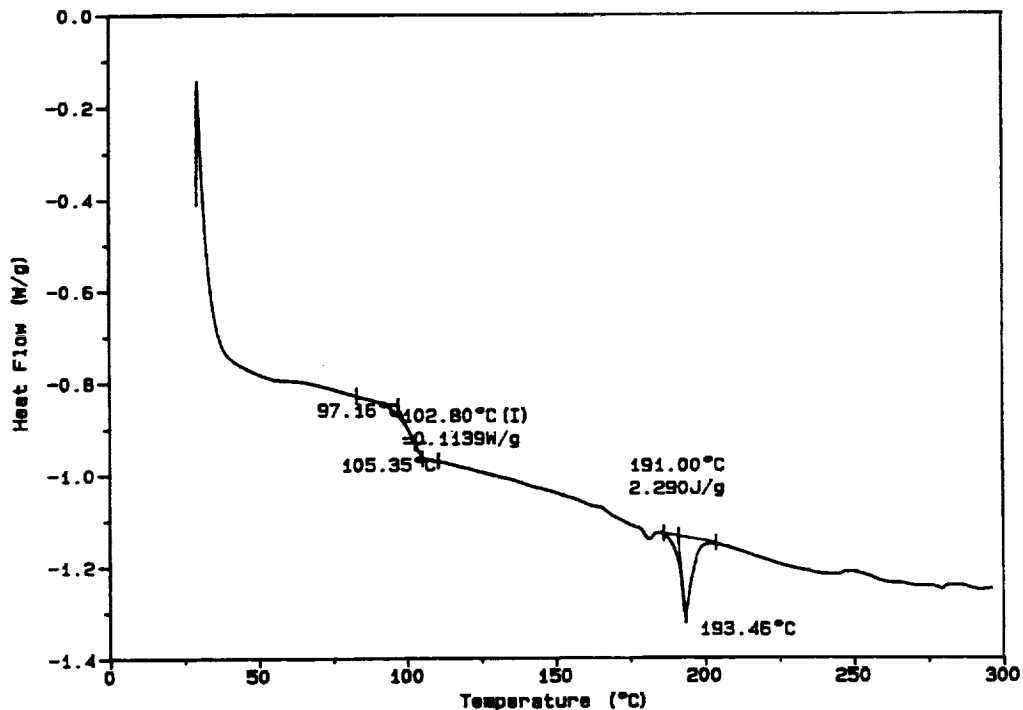


Figure 4. Polystyrene temperature variation curve indicating melting point of 193°C

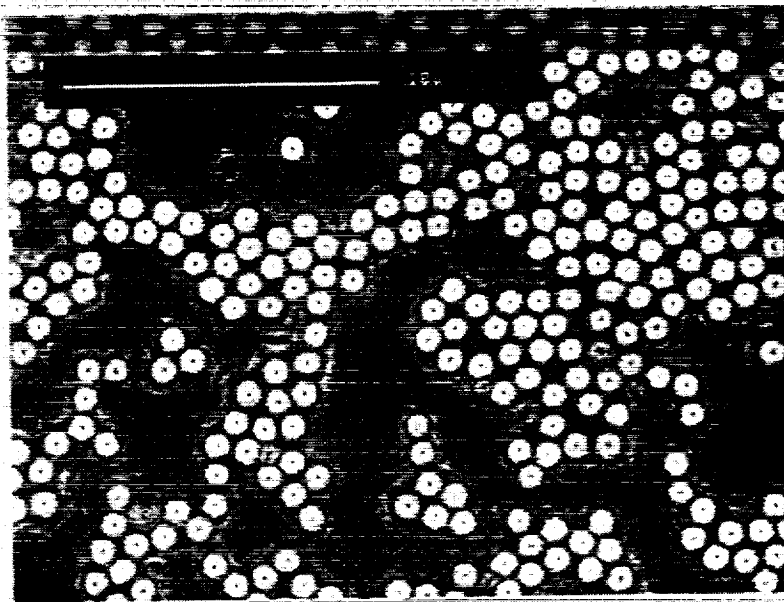


Figure 5. Monodisperse spherical polystyrene beads of 0.95 μ

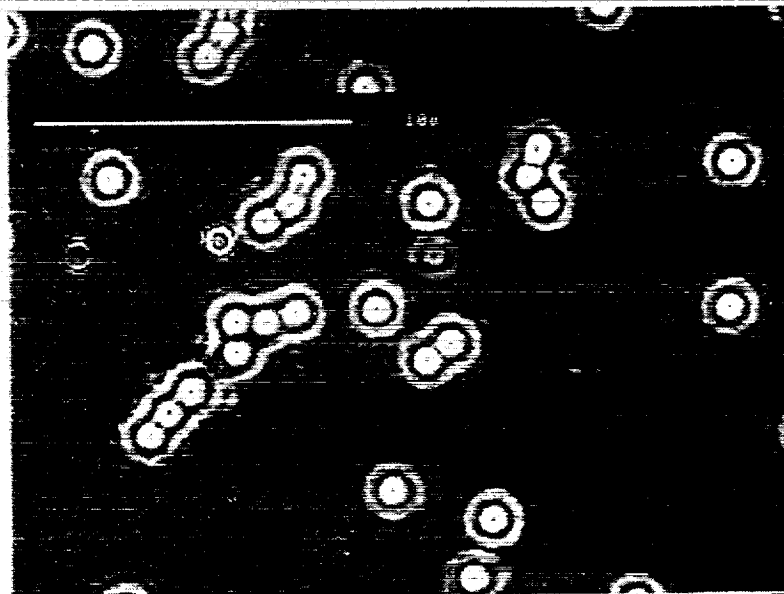


Figure 6. Monodisperse, micro spheres cross-linked polystyrene beads

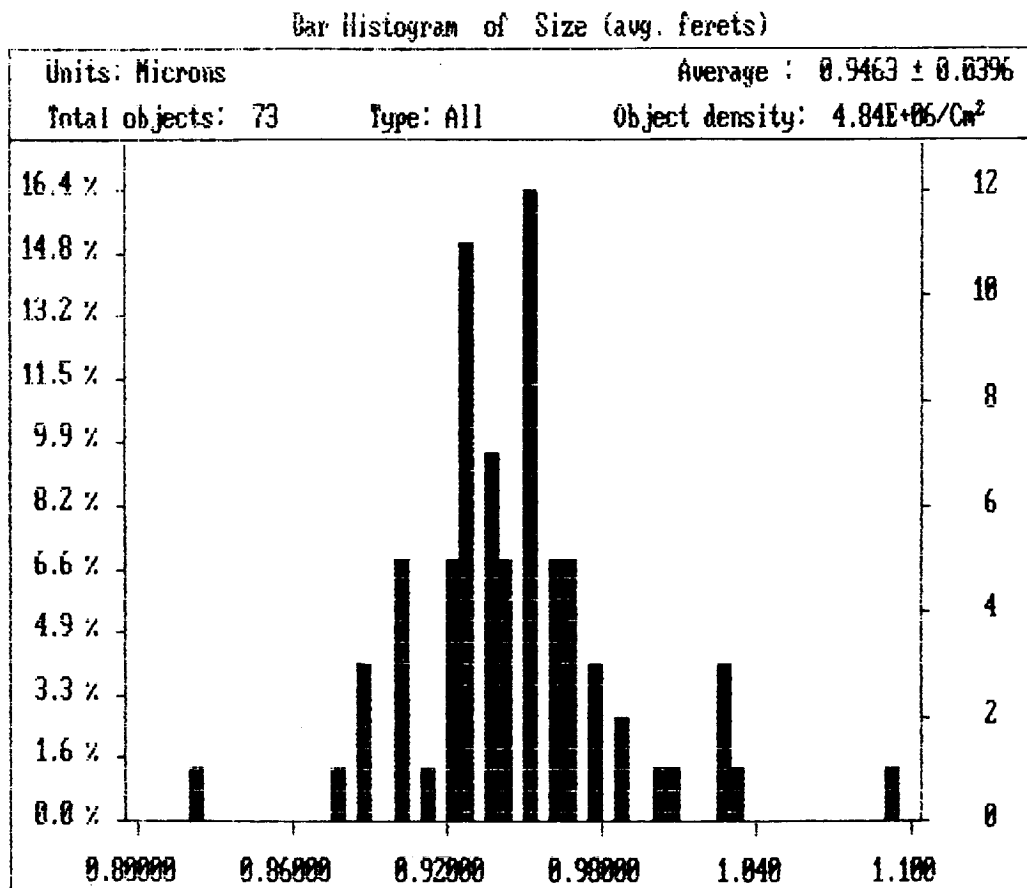


Figure 7. Percent of various size beads produced, illustrating monodispersity beads size 0.95μ (on average)

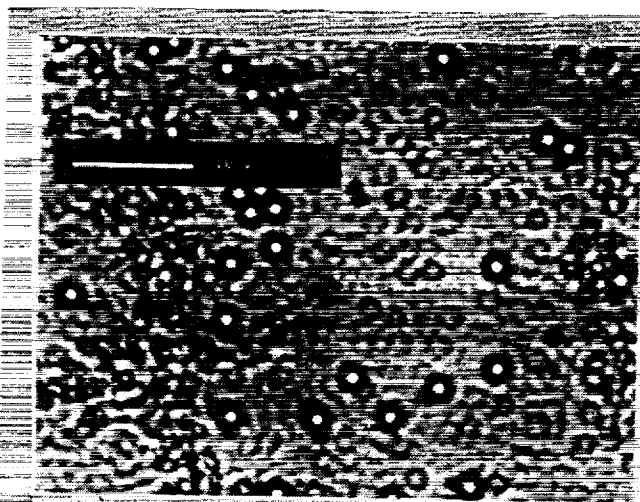


Figure 8. A 22-hour sample showing few particles

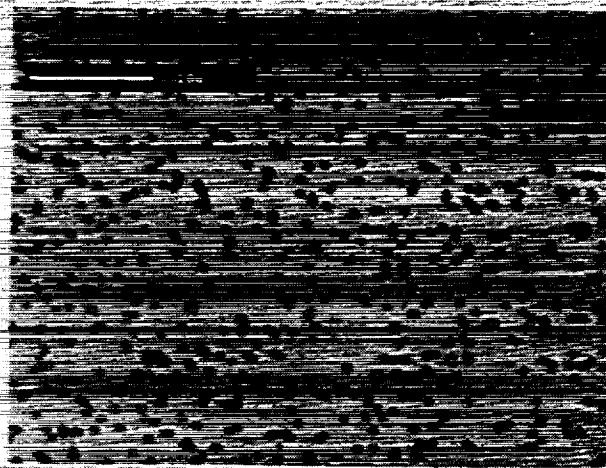


Figure 9. A 4-hour sample showing no particles

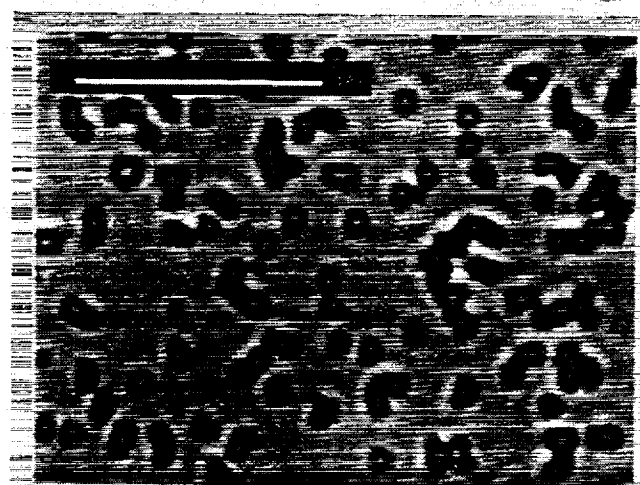


Figure 10. Wormlike formation of cross-linked polystyrene after 6 hours

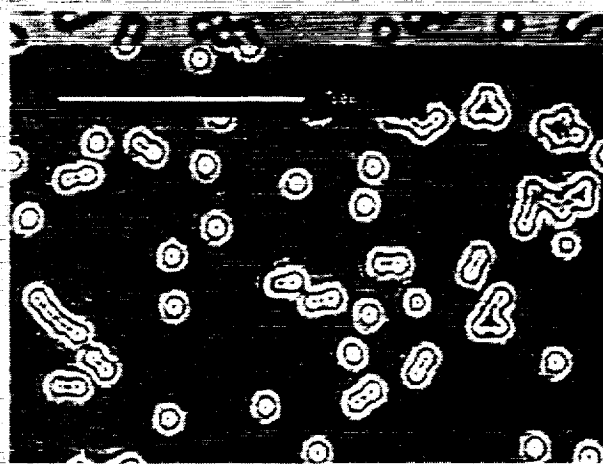


Figure 11. Monodisperse, spherical cross-linked polystyrene of size $0.7\ \mu$

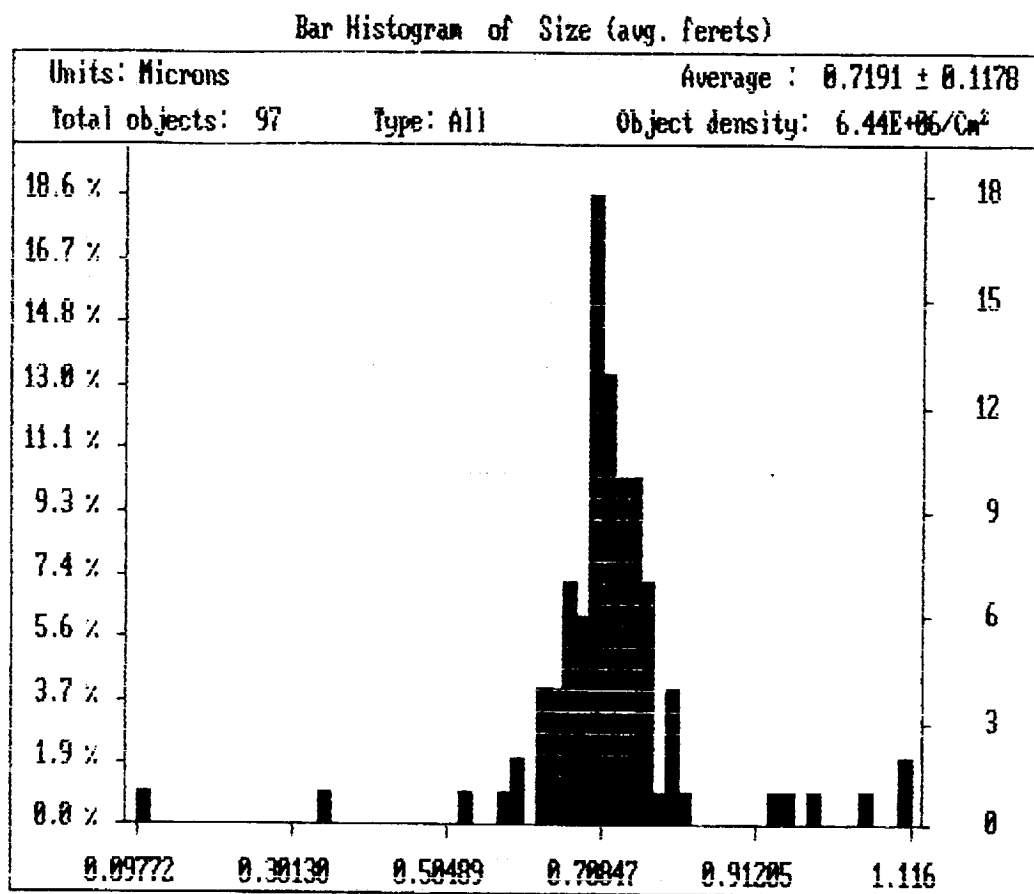


Figure 12. Percent of various size beads produced, illustrating monodispersity of bead size 0.7μ (on average)